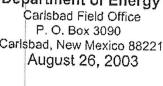


## Department of Energy

P. O. Box 3090 Carlsbad, New Mexico 88221 August 26, 2003







Dr. Amrit S. Boparai Analytical Chemistry Laboratory Argonne National Laboratory-East 9700 South Cass Avenue Argonne, IL 60439-4837

RE:

Evaluation of Corrective Action Plans Argonne National Laboratory-East (ANL-E) CARS 03-065 and 03-066 for Audit A-03-18

Dear Dr. Boparai:

The Carlsbad Field Office (CBFO) has evaluated the Corrective Action Plans for CARS 03-065 and 03-066. The remedial actions, investigative action, root cause and actions to prevent recurrence for CAR 03-065 were not acceptable. The remedial action for CAR-03-066 was acceptable; however, verification of the remedial action based on the review of documentation submitted with the CAP on August 7, 2003 was not acceptable. The evaluation results are documented on the enclosed CAR Continuation Sheets.

If you have any questions or comments, please contact me at (505) 234-7442.

Sincerely,

M. Lea Chism

Quality Assurance Specialist

## Enclosure

cc: w/enclosure A. Holland, CBFO \*ED L. Chism, CBFO K. Watson, CBFO \*ED M. Brown, CBFO \*ED K. Joshi, DOE-CH \*ED A. Gable, DOE-CH \*ED M. Eagle, EPA \*ED S. Zappe, NMED \*ED B. Walker, EEG \*ED D. Gill, CTAC \*ED A. Arceo, CTAC \*ED C. Watkins, CTAC \*ED A. Pangle, CTAC \*ED K. Dunbar, WRES CBFO QA File

\*ED denotes electronic distribution CBFO:QA:MLC:GS:03-2546:UFC 2300.00

**CBFO M&RC** 



## CBFO CORRECTIVE ACTION REPORT

(Continuation Sheet)

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1. CAR No. 03-065	*****	2. Activi	ty Report No. A-03-18		3. Page	1	Of	1
Block #17 Evaluation of Corrective Actions Proposed by the Responsible Organization:								
The response submitted by Argonne National Laboratory on August 7, 2003 is rejected. The response is rejected based on the following:								
1. a. SW-846, Chapter One, section 4.6, Calibration Records & Traceability of Standards/Reagents states that "Calibration is a reproducible reference point to which all sample measurements can be correlatedThe accuracy of the calibration standards is important because all data will be in reference to the standards used".								,
b. SW-846, Metho of the relationsh into the instrument	b. SW-846, Method 8000B, section 7.4 states that "Initial calibration of an analytical instrument involves the delineation of the relationship between the response of the instrument and the amount or concentration of an analyte introduced into the instrument In order to perform quantitative measurements, this relationship must be established prior to the analysis of any samples"							
Conclusion: The analytical scheme ANL-E is proposing does not comply with the above requirements. The site proposes to use calibration standards as the "unknown" in the scheme and the PDP samples as the reference point. This is not acceptable.								
<ul> <li>a. SW-846, Method 8260B, section 5.7 states that stock standards "may be prepared from pure standard materials or purebased as certified solutions". A standard that has expired can no longer be regarded as a "certified" standard.</li> <li>b. SW-846, Method 8260B, section 5.12 states "Calibration standards — There are two types of calibration standards used for this method; initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations".</li> </ul>								
Conclusion: Expired standards cannot be used for calibration as they are no longer "certified".								
The remainder of ANL-E's response was not assessed as it is based on the premise of calibration using expired standards. This practice is not acceptable.								
Part of the mission of ANL-E, with regard to the PDP program, is to verify that the concentration of analytes in the PDP samples is correct. It is unclear how the laboratory would do this when they propose using expired standards for calibration and the PDP samples as the only "knowns" in the analytical scheme.								
It is acceptable for the laboratory to utilize a documented protocol to verify expired standards so that they may be used beyond the original expiration date. However, this analytical scheme must be based on valid instrument calibration using unexpired, certified standards.								
Evaluated By: Dorothy G Name	2011	***************************************	8 /21 /03 Date					
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## CBFO CORRECTIVE ACTION REPORT

(Continuation Sheet)

1. CAR No. 03-066	2. Activi	ty Report No. A-03-18	3. Page 1 Of 1				
Block # 17 Evaluation of Corrective Actions Proposed by the Responsible Organization and Verification of Corrective Action Documentation:							
The response submitted by Argonne National adequacy issues identified in this CAR; howe identified. Verification of corrective action is			168, Rev. 5 to address the c. 6, did not address the issues				
CAR item A: The initial calibration section (8.4.1) does not Response: "Section 8.8.1 of the revised SO	describe l P, ACL 16	ow retention time and retention time windows, Rev. 6, addresses item A given above".	vs are determined.				
Verification of Corrective Action: Section 8. windows on an annual basis. Other circumstant should initiate RTW determination are not add		revised SOP, ACL 168, Rev. 6 only requires as major instrument maintenance or installati	determination of retention time on of a new analytical column, that				
<u>CAR item B:</u> The procedure does not require generation of items.	method pre	ecision and accuracy data.					
Response: "Section 10 of the revised S	OP, ACL	68, Rev. 6, addresses Item B given above".					
Verification of Corrective Action: Section 10 of the revised SOP, ACL 168, Rev. 6 reads: "Method Performance The laboratory has demonstrated it's ability to meet the program required MDLs". This CAR item deals with precision and accuracy data, not MDLs.							
CAR item C: The procedure does not describe what records		¥)					
Response: "Section 9.6 of the revised S	OP, ACL	168, Rev. 6, addresses item C given above".					
Verification of Corrective Action: Section 9.6 Refer to Work Plan for Headspace Gas Perform Section 9.6 appears to only address raw data rastate what information, associated with the CA information.	than than I	ionstration Frogram Sample Preparation, Ve	rification, and Distribution".				
CAR item D: The procedure does not address the holding ten nonitoring the temperature in the area for stora	iperature r ge of PDP	equirement for VOCs SUMMA canister, her and calibration canisters.	ice there was no documentation for				
Response: "Section 7 of the revised SOF	, ACL 16	3, Rev. 6, addresses item D given above".					
Verification of Corrective Action: Section 7 of the revised SOP, ACL 168, Rev. 6 does not address the holding temperature requirement or headspace gas samples. Additionally, the response did not specify if temperature monitoring of the storage area will be required.							
Evaluated By: Donothy Gill	,						
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Name		Date	*				
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